Methods of ferrite grain determination test for steel

Introduction This Japanese Industrial Standard was partially revised in 1977 since establishment in 1957, and has come to present. Though nearly 20 years have elapsed from the last revision, since there is a necessity for achieving conformance to International Standard ISO 643:1983, Steels — Micrographic determination of the ferritic or austenitic grain size cojointly with technical progress during that time, the revision is executed.

Main revised points are as follows.

- a) The application range is conformed to 0.25 % or under carbon content.
- b) The use symbol and the relation formula are conformed to International Standard. Further, the item of report is newly established.
- c) As the judging method, a counting method and a judging method by intersected segment (grain size) are added as Annexes.
- 1 Scope This Japanese Industrial Standard specifies the testing method of measuring ferrite grain size (hereafter referred to as "grain size") of steel mainly with a carbon content not more than 0.25 %.

Remarks: The corresponding International Standard to this Standard is shown as follows.

ISO 643 Steels — Micrographic determination of the ferritic or austenitic grain size

- 2 Definitions For the purpose of this Standard, the following definitions apply:
- a) grain size Grain size means the size of ferrite crystal grain of steel, and its marking is expressed in grain size number.
- b) grain size number The grain size number means the number expressed in accordance with either the method 1) or 2) undermentioned after the grain size has been measured by the specified method.
 - Table 1 shall apply to the expression of grain size number when the measurement is made by comparison method.

Table 1 Grain size number

Grain size No.	Number of crystal grain per mm² of sectional area m	M an sectional area of crystal grain mm²	Mean number of crystal grain in 25 mm² at 100 magnifications n
-3	1	1	0.062 5
-2	2	0.5	0.125
_1	4	0.25	0.25
0	8	0.125	0.5
1	16	0.062 5	1
2 .	32	0.031 2	2
3	64	0.015 6	4
4	128	0.007 81	8
5	256	0.003 90	16
6	512	0.001 95	32
7	1 024	0.000 98	64
8	2 048	0.000 49	128
. 9	4 096	0.000 244	· 256
10	8 192	0.000 122	512

Remarks: In Table 1, the following relation formula is concluded. $m=8\times2^{G}$

2) In measurements made by intercept method, the following formula shall apply to the expression of grain size number. The grain size number shall be rounded off to the first decimal place.

$$n=500(M/100)^2(I_1\times I_2)/(L_1\times L_2)$$
 $G=(\log n)/(0.301)+1$ (1)

where, G:g

G: grain size No.

n: number of grain size in 25 mm² under a microscope of 100 magnification

M: microscope magnification

 $L_1(\text{or } L_2)$: total length (unit: mm) of one linear length of the

segments orthogonally crossing each other

 I_1 (or I_2): total number of crystal grain intercepted by L_1 (or

 L_2

Remarks: Attached Fig. 3 shows a relationship included within formula (1) in a graphic chart.

- c) mixed grain Mixed grain shall mean the presence of 20 percent or more of crystal grain different, approximately 3 or more in grain size number within one visual field.
- d) elongation rate This is the ferrite crystal grain elongated due to the working, which shall be calculated from the following formula:

 $e=n\sqrt{n_2}$

wher,

e : elongati n rate

 n_1 : number of crystal grain intercepted by a certain length of a segment orthogonal to the direction in which the grains have been elongated

 n_2 : number of crystal grain intercepted by a segment identical in length to the n_1 segment parallel to the direction in which the grains have been elongated

- 3 Classification of testing methods Testing methods shall be classified into following two categories:
- a) Comparison method
- b) Intercept method

The test shall be made, usually, by comparison method, but, where ferrite crystal grains are markedly elongated or required to be accurate, intercept method should be rather used.

Further, the counting method may be used as agreed upon between the purchaser and supplier. In this case Annex 1 applies. Also, the intersected segment method may be used as agreed upon between the purchaser and supplier. In this case, Annex 2 applies.

4 Treatment of test piece Steel with the section parallel to or at a right angle to the working direction shall be etched after being finished by polishing. For this etching, a solution, preferably of about 5 percent nitric acid alcohol should be applied for 15 s. If necessary, the surface, as it is, of the test piece may be subjected to the testing.

Remarks: As the etching solution nitryl (ethanol nitrate), picryl (ethanol picrate) or an appropriate reagent may be used excepting this.

5 Testing methods

- 5.1 Comparison method The grain sizes as appearing on the etched surface shall be observed under a microscope and compared with a standard figure.
- 5.2 Intercept method The number of the ferrite crystal grain intercepted by the 2 segments of a fixed length orthogonally crossing each other shall be determined by observing the grains appearing on the etched plane under a microscope or by means of a microphotograph.

However, the ferrite crystal grain partially intercepted at both ends of a segment shall be counted only one grain and in case there is one partially intercepted grain at one end of a segment, no count shall be taken.

Also, the magnification of the microscop shall be so adjusted that the number of ferrite crystal grain to be intercepted by one segment will be not less than 10 within one visual field and the measurement shall be continued for a veral visual field until those grains amount to not less than 50.

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Remarks: For the m thod for observing with a microscope, there are microphotograph and obs rvation on ground glass excepting visual observation.

6 Method of determining grain size in each visual field

6.1 Comparison method

- 6.1.1 The grain size measured under a microscope shall be compared with the standard figure in Attached Fig. 1 to determine a corresponding grain size number. As a rule, the microscope in this case shall have a magnification of 100, the actual visual field shall be a circle of 0.8 mm diameter, and the size and shape of the projected image or the photographic print shall be a circle of 80 mm diameter.
- 6.1.2 For grain sizes falling between two consecutive grain size numbers, the smaller grain size number shall be used, but the number of 0.5 shall be added to that number.
- 6.1.3 When it is difficult to make a determination under a microscope of 100 magnification, 50 or 200 magnification may be used. However, at that time, as given in Table 2, for example, at 50 magnification, the grain size number, thus obtained shall be decreased by 2, and at 200 magnification, the grain size number shall be increased by 2.

Table 2 Relation of grain size number to ordinary magnification

Magni- fication of image	Grain size number related to image to be discriminated with standard drawing number							
	1	2	3	4	5	6	7	8
50	-1	0	1	2	3	4	5	6 8
100 200	3	4	3 5	6	7	8	9	10

- 6.1.4 For mixed grains, the proportion of the areas of each grain size number shall be determined by eye.
- 6.1.5 In the case where pearlite and the like are mixed in great numbers so far as the mixed state is banded or granular, the area proportion of the mixed structure and of ferrite crystal grain shall be determined by eye. Subsequently, determination of a corresponding grain size number for only the ferrite crystal grain shall be made by comparison with the standard figure in Attached Fig. 1.

Remarks: Attached Fig. 2 indicates the presence of a pearlite phase in the mixture.

6.2 Intercept method

6.2.1 The grain siz number shall b d termined by f rmula (1) or Attached Fig. 3 on the basis of the number of ferrite crystal grain measur d und r a micr scope.

6.2.2 In the cas where the p arlite and the lik are mixed in great numbers, the area proportion of the mixed structure and of ferrite crystal grain shall be determined by an appropriate m thod (1) and then the number of crystal grain in square 25 mm² at 100 magnification shall be measured by intercept method. The results shall be converted into the number of ferrite crystal grain per 25 mm² square to obtain the grain size number determined by formula (1) or by Attached Fig. 3.

Note (1) For example, point counting method, gravimetric method, phototube method, linear analysis method, etc.

6.3 The judging method by a counting method may be used as agreed upon between the purchaser and supplier. In this case, Annex 1 applies.

7 Method for synthetic determination

7.1 In comparison method, the mean grain size number which derives from the method of determining grain size in each visual field in 6.1 by the formula as shown below shall be taken as the grain size of the steel. The mean grain size number shall be rounded off to the first decimal place.

The number of visual fields observed shall be, as a rule, from 5 to 10. Table 3 shows an example.

$$g = \frac{\sum (a \times b)}{\sum b}$$

where,

g: mean grain size number

a: grain size number in each visual field

b: number of visual fields indicating the identical grain size number

Table 3 Calculation of grain size (example)

Grain size No. in each visual field	Number of visual fields	axb	Mean grain size No. g	Grain size
6	2	12	$\frac{66}{10} = 6.5$	6.5
6.5	6	39	10	1
. 7	2	14		
Total	10	65		

7.2 In intercept method, the grain size number obtained by the method under 6.2 shall be taken as the grain size of the steel.

7.3 For mixed grain under 6.1.4, the ratio of the mixture shall be determined by the synthetic mean values of the proportion of area of each grain size number.

7.4 The judging method by the intersected segment (grain size) may b us d as agreed upon b tween the purchaser and supplier. In this case, Annex 2 applies.

8 Marking

8.1 Marking shall b made on the basis of the results of synthetic determination in order of the symbol(2) for the type of testing m thod, the symbol(3) for the tested plane, the grain size, and the number of visual fields, as given below.

Notes (2) Symbols for the types of testing methods shall be as follows:

FGc: comparison method

FGt: intercept method

(3) Symbols for the planes tested shall be as follows;

V: vertical cross section

P: parallel section

S: surface

Example 1 For not mixed grain

FG_C-V3.5₍₁₀₎............ (A grain size of 3.5 in vertical cross section derived by synthetic determination obtained from 10 visual fields by Comparison Method as outlined in 5.1.)

FG_t-P6.5..... (A grain size of 6.5 in a parallel section derived by Intercept Method as outlined in **5.2**.)

FG_C-V3.5₍₁₀₎.......... (Mixed with 20 % banded pearlite) (A grain size of 3.5 in a parallel section mixed with 20 % banded pearlite derived by synthetic determination obtained from 10 visual fields by Comparison Method as outlined in 5.1.)

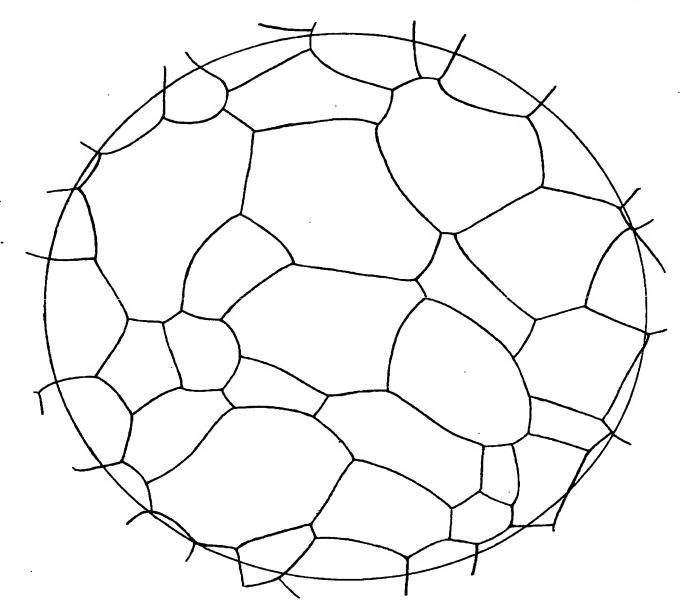
Example 2 For mixed grain

FG_C-V[3(70%)+6(30%)]₍₁₀₎(70 % of grain size of 3 and 30 % of grain size of 6 in vertical cross section, derived by synthetic determination obtained from 10 visual fields of mixed grains by Comparison Method as outlined in 5.1.)

8.2 For elongated crystal grain, the elongation rate, if necessary, shall also be noted, as given below.

Example e-P 2.3(An elongation rate of 2.3 in a parallel section)

- 8.3 If necessary, the location of the tested planes shall also be noted.
- 9 Report When a test report is required, the following items as report items shall be selected as agreed upon between the purchaser and supplier.
- a) Symbol of kind of tested steel materials
- b) Test method (symbol according to the test method)
- c) Test conditions
- d) Grain size number

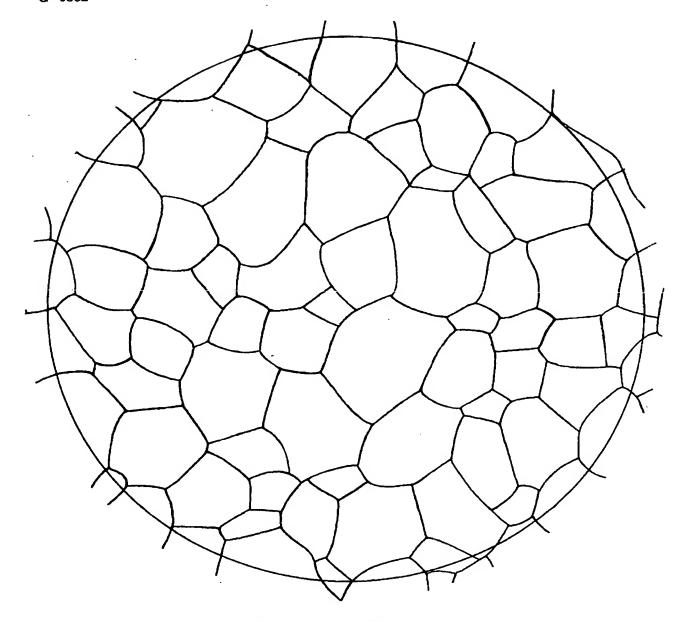


Grain size No. 1

Attached Fig. 1 Standard figure of ferrite grain size (100 magnifications)

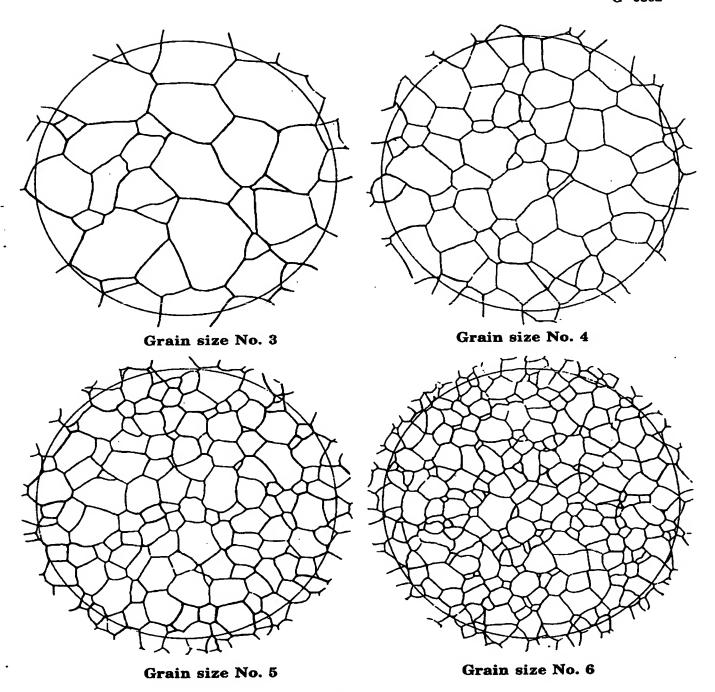
These standard figures are shown for reference on a reduced scale of 70% of original figure in this handbook.

In the case of practical examination, refer to the original Standard.

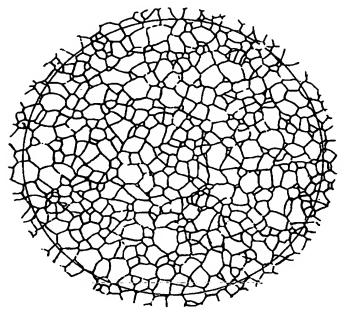


Grain size No. 2

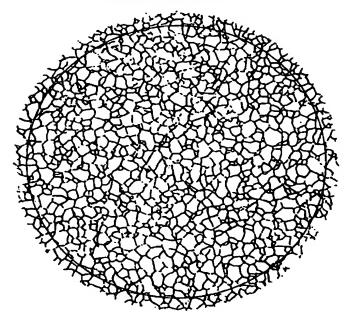
Attached Fig. 1 (continued)



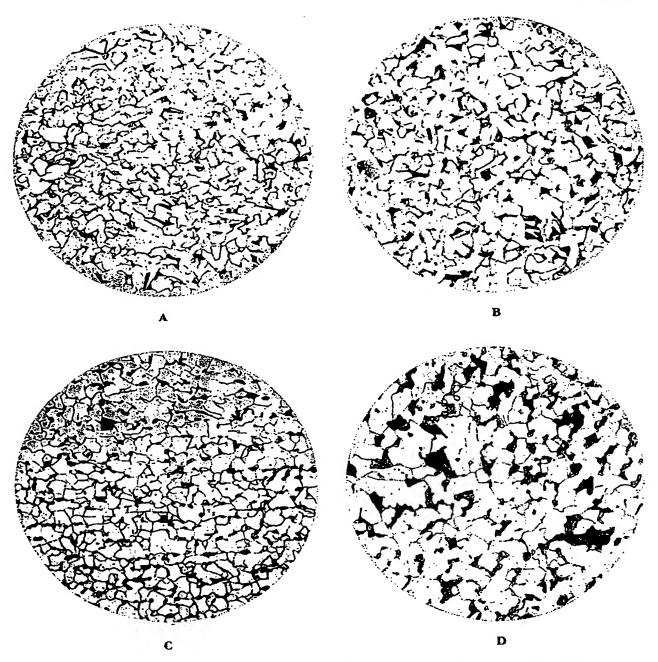
Attached Fig. 1 (continued)



Grain size No. 7



Attached Fig. 1 (concluded)



Attached Fig. 2 Informative figure of microstructures mixed with pearlite (100 magnifications)

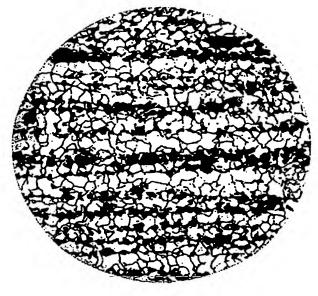
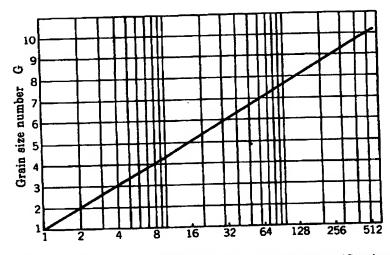


Photo identifi- cation No.	Pearlite area %	Grain size	Re- marks
Α	5.9	7	Granular structure
В	8.8	6.5	Granular structure
С	8.3	7	Band structure
D	15.2	6	Granular atructure
E	20.4	7.5	Band structure

E

Attached Fig. 2 (concluded)



Number of crystal grains per 25 mm² n (at 100 magnifications)

Attached Fig. 3 Relationship of grain size number and the number of crystal grains

Ann x 1 (normative) Counting m thod

Introduction This Annex has been prepared based on the counting method specified in ISO 643:1983, Steels — Micrographic determination of the ferritic or austenitic grain size without changing technical contents.

Further, the places to which the dotted underlines are applied in this Annex are items added to the original International Standard.

1 Scope This Annex 1 specifies the measuring method for obtaining a crystal grain size.

2 Measuring method

2.1 Measurement A particle size is measured with a microscope as follows.

The size of the visual field to be observed in an area enclosed with a 79.8 mm diameter circle (area 5000 mm²) traced on the ground glass screen of the microscope (or on the photomicrograph).

Further, the measurement can be executed by the same method by use of the area nclosed by the square of 70.7 mm side length (the area equal to 5000 mm^2) or the rectangle of the same area of, for example $80.0 \text{ mm} \times 62.5 \text{ mm}$.

The linear magnification g of the image shall be such that at least 50 crystal grains can be counted in the observing visual field, and shall be generally 100.

In this case, the actual surface area on a test specimen is 0.5 mm² (see Annex 1 Fig. 1).

The number of crystal grains n_1 , completely inside the circle and the number of crystal grains n_2 intersected by the circumference shall be counted.

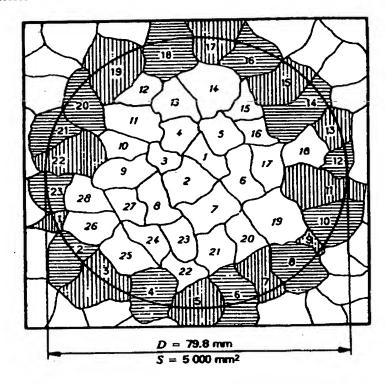
2.2 Judging method The judging is carried out by calculating according to from formula (1) to formula (7) from the number of crystal grains n_1 , enclosed completely in the measured circle and the number of crystal grains n_2 intersected by the circumference.

 The mean crystal grain area (mm²) $a=1/m \qquad (7)$

A calculated value m corresponds to each particle size number G. The particle size number is obtained within the range stated on Annex 1 Table 1.

2.3 Marking The marking shall be as described in 8 of the text. However, the symbol (1) shall be as follows.

Note (1) FG (Count): counting method



Annex 1 Fig. 1 Evaluation of number of grains in an area enclosed by a circle

Annex 1 Table 1 Grain siz number

Grain	Number of crystal grains per 1 mm ² sectional area			
size number G	Nominal value		values to (incl.)	
-3	1	0.75	1.5	
-2	2	1.5	3	
-1	4	3	6	
0	. 8	6	12	
1	16	12	24	
2	32	24	48	
3	64	48	- 96	
4	128	96	192	
5	256	192	384	
6	512	384	768	
7	1 024	768	1 536	
8	2 048	1 536	3 072	
9	4 096	3 072	6 144	
10	8 192	6144	12 288	

Annex 2 (normative) Determination method by intersected segment (grain size)

Introduction This Annex has been prepared based on the determination method by intersected segment (grain size) specified in ISO 643:1983, Steels — Micrographic determination of the ferritic or austenitic grain size without changing technical contents.

Further, places to which the dotted underlines are applied in this Annex are items added to the original International Standard.

1 Scope This Annex 2 specifies the determining method by the intersected segment (grain size).

Remarks: The number of crystal grains intersected with a measuring line shall be counted on the ground glass screen of a microscope or on the microphotograph within a representative visual field. Since the same result is obtained by both methods, the intersected segments can be counted. The measuring line may be either of straight line or circle. The measuring grid of Annex 2 Fig. 1 indicates the kind of the measuring line to be used. The grid is only once applied to one visual field to be investigated. In ord r to count effectiv ly, it is applied at random to a suffici nt number of visual fields.

- 2 Classificati n f test methods
- a) Linear intersected segment method
- b) Circular intersected segment method

3 Test method

3.1 Linear intersected segment method The measuring line consists of four straight parts with a total length of 500 mm arranged as shown in Annex 2 Fig. 1. This arrangement allows the effect of grain anisotropy to be reduced. The vertical and horizontal lines are used for measuring the grain size in the different directions. One of the two lines is oriented in the desired direction.

The magnification shall be selected so that at least 50 intercepts are obtained in any one measurement.

The following rules shall be followed:

3.1.1 In the case where the intersected segments are counted When the measurement line terminates inside a grains, the segment intersected by this grain is counted as one half.

3.1.2 In the case where the intercepts are counted

- a) The end of the measuring line is counted as 1/2 only when it exactly touches a grain boundary;
- b) When the line is tangential to the grain boundary, one intercept is also counted;
- When the intercept coincides with a triple point (junction of 3 grains) it is counted as 1.5 intercepts;
- d) In the case of grains of irregular shape when the line bisects the same grain at two different points, the two intercepts shall be counted.
- 3.2 Circular intersected segment method The measuring line consists either of a set of three concentric circles shown in Annex 2 Fig. 1 or one single circle.

The total length of the three circles of the grid shown in Annex 2 Fig. 1 is 500 mm. The magnification shall be selected so that there are at least 50 intercepts when the measurement grid is superposed on the field examined.

In the case of a single circle, the largest circle with a circumference of 250 mm is used. In this case, the magnification to be used shall enable at least 25 intercepts to be counted.

The circular intersected segment method tends to give slightly high intersected segment values and thus a slightly low number of intercepts. In order to compensate for this, the intercepts caused by a triple point shall be counted as two intercepts instead of 1.5 as is the case with the linear intersected segment method.

4 Calculating method By repeating the measurements of the number of intercepts on different fields several tim s, it is possible to arrive at the mean value of the number of intercepts \bar{N} .

If L is the length of the measurement line, the mean number of intercepts per 1 mm is

1

$$\bar{N}_L = \bar{N}/L$$

The mean value of the inters ct d segm at is giv a by the formula

$$\overline{L}=1/\overline{N}_L$$

In the case of non-equiaxial structures, it is possible to determine the number of intercepts on three sections respectively:

- 1 longitudinal section;
- 2 1 transverse section;
- 3 1 perpendicular section.

The mean number of intercepts per millimetre, \overline{N}_L , is then determined by the formula.

$\overline{N}_L = (N_X + N_Y + N_Z)/3$

where, N_x ; mean number of intercepts per millimetre on the longitudinal section

 $N_{\rm Y}$: mean number of intercepts per millimetre on the transverse section

 N_z : mean number of intercepts per millimetre on the perpendicular section

- Remarks 1 Grains of different size indices In certain cases, the surface examined may include grains belonging to two or more different systems of size indices. This can be recognized by the presence of several grains of greatly different dimensions from those of the whole, for example. This may then lead to making counts by dimensions and to determining two or several indices with a possible mention of their frequency or their position.
 - 2 Twin grains Unless otherwise specified, these are counted as a single grain (see Annex 2 Fig. 2).
 - 3 Non-equiaxed grains The designation of the grain size may be completed by determining the ratio of their dimensions obtained by measuring on two suitably selected rectangular axes, one of which may be that of the rolled product, the length intersected by a similar number of grains and taking the ratio of these lengths.
- 5 Determination method The determination of the measured results shall be as follows. Indices FG (ASTM)=0 corresponds to 32.0 mm measured with 100 magnifications.

The indices FG (ASTM) are respectively obtained from the following formulae form average intersected segment (\overline{L}) or average number of intercepts (\overline{N}_L) for unit length (mm).

Indices FG (ASTM)=-3.287 7-6.643 9 $\log_{10} \overline{L}$ Indices FG (ASTM)=-3.287 7+6.643 9 $\log_{10} \overline{N}_L$ 6 Marking The marking shall b as given in 8 of th text. However, the grain size number(1) shall be as follows.

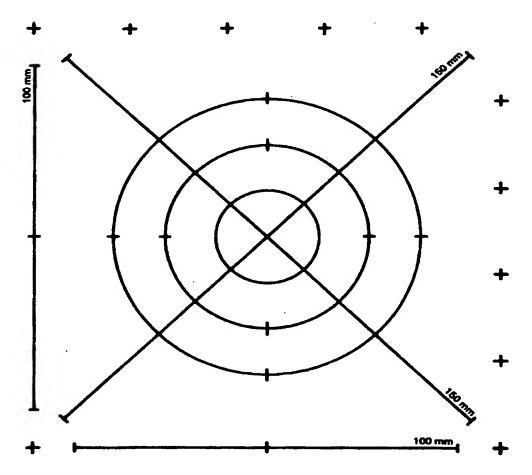
Note (1) FG (ASTM): th indices FG (ASTM) determined with intersected segm at (gain size)

The dimensions in millimeters of the three circles

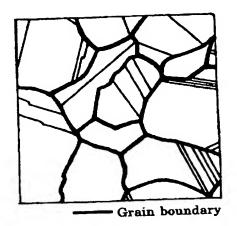
Unit: mm

Diameter	Circumference		
79.58	250.0		
53.05	166.7		
26.53	83.3		

Total 500.0



Annex 2 Fig. 1 Measurement grid for the intersected segment method



Annex 2 Fig. 2 Evaluation of number of grains (twin grains)